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On Deformation Behaviour of Polycrystalline Iridium at Room Temperature

How structure rules by mechanical properties

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Deformation and fracture behaviour of cold drawing iridium wire under tension at room temperature is examined. High purity polycrystalline iridium was manufactured using pyrometallurgical technology. During the initial stage of cold rolling, iridium wire has its usual grain structure and exhibits brittle deformation behaviour: poor plasticity and brittle transgranular fracture (BTF). However, the wire begins demonstrating high plasticity including necking in spite of the brittle fracture mode when the lamellar structure has been formed in iridium during cold drawing.

1. Introduction

The platinum group metal iridium is perhaps the most puzzling metal on Earth due to its property of being cleavable and a plastic solid simultaneously (1). This refractory face-centred cubic (fcc) metal (T_{melt} = 2446°C) serves as the structural material for applications under extremely hard conditions (2, 3) such as containers for fuel sources in radioisotope generators for deep space missions (4), or crucibles for growing oxide crystals for power lasers (5). Industrial technology for refining and processing iridium, based on traditional chemical refining methods (2, 6, 7), has been developed over the past 60 years (8–10). Based on these achievements, it has been shown that polycrystalline iridium exhibits limited plasticity due to intergranular fracture at room temperature, but its plasticity increases considerably under elevated temperatures (9–12). The segregation of non-metallic impurities on the grain boundaries was considered the cause of poor workability of polycrystalline iridium (12). This type of deformation behaviour agrees with empirical knowledge on deformation and fracture of metals (13, 14). On the other hand, single crystalline iridium behaved unusually: it cleaved under tension after considerable elongation (13, 15), but never failed under compression (16–18). At room temperature the fracture mode of iridium single crystals was attested as BTF (1, 15), while brittle intergranular fracture (BIF) was the fracture mode for polycrystalline iridium (11, 19). Analysis of the causes of cleavage in iridium has shown that it satisfies some empirical cleavage criteria (18–20) due to features in the elastic moduli in comparison with other fcc metals (18, 21). This fact leads to the conclusion that the inclination to cleavage is an intrinsic property of iridium, whereas impurities only reinforce it (18–20, 22, 23). However, the analysis of interatomic bonding in iridium has shown that BIF may also be considered as the intrinsic fracture mode of polycrystalline iridium (24).

The pyrometallurgical scheme for the refining of iridium, including: (a) oxidation induction melting; (b) electron beam melting; and (c) growing massive single crystalline workpieces by electron beam, became an alternative technology to manufacture 'plastic' iridium (25–27). Pyrometallurgical iridium demonstrated considerable plasticity prior to failure under tension in both the single crystalline state (28) and the polycrystalline state (29), while its elastic properties were the same as findings obtained earlier (30). It was confirmed that the intrinsic fracture mode of this 'plastic' iridium is BTF, while BIF is induced by harmful non-metallic impurities such as carbon and oxygen (31, 32). Indeed, the portion of BTF on the fracture surface of plastic iridium is considerably higher than BIF (33, 34). Deformation mechanisms and, hence, behaviour of pyrometallurgical iridium were the same as a normal fcc metal excepting the special fracture mode (35–38). Recent studies of deformation and fracture behaviour of iridium have shown that new participants achieved the technological level that allows 'plastic' iridium to be manufactured (39, 40), including iridium single crystals (41). Also, the old problem concerning the intrinsic fracture mode of polycrystalline iridium or the competition between BTF and BIF in iridium remains (42). Therefore, in the present paper, the deformation and fracture behaviour of iridium wires under tension at room temperature are considered in light of the discussion on this problem.

2. Materials and Methods

Pyrometallurgical iridium was used in this work. It was high purity metal, free of non-metallic contaminants such as carbon and oxygen. The refining procedure and, hence, impurities content were the same as the metal used in earlier work by our group: non-metallic elements <0.1 ppm; tungsten, molybdenum, niobium, iron, zirconium, copper, gadolinium, yttrium, gallium, nickel, palladium, zinc, magnesium, calcium –0.1–1 ppm; platinum, rhodium ~10 ppm (26, 27, 29, 38). Experience has shown that pyrometallurgical refining could be limited by the first and second procedures without loss of quality of the metal. Therefore, the operation of the growth of the massive single-crystalline iridium workpieces was not carried out in this work. The mechanical treatment of the pyrometallurgical iridium included: (a) forging the ingot into sheet at 1500–2000°C in air; and (b) rolling the sheet at ~800°C in air. The resulting metal could be processed like platinum. The cold drawing iridium wire, whose diameter varied from 2.7 mm to 0.5 mm, was prepared from this plastic metal. No long-term recrystallisation annealing of this wire was carried out because this procedure leads to embrittlement and failure of iridium wire due to BIF. Tensile testing was carried out with the help of an Autograph AG-X 50N tensile/compression tester (Shimadzu Corporation, Japan) (traverse rate of 1 mm min^{-1}) at room temperature. The lengths of working parts of iridium wire samples were 100 mm. The structure of the samples before and after testing was examined by conventional X-ray diffraction (XRD) technique on the D8 Advance diffractometer (Bruker Corporation, USA) with copper kα irradiation. Back surfaces of each sample before and after testing were documented on a light metallographic microscope. The fracture surfaces of samples were studied on the scanning electron microscope JSM-6390 (JEOL Ltd, Japan).

3. Results

The first set of iridium samples consisted of 10 pieces taken from a commercial parcel of cold drawing thin iridium wire produced by UralInTech (Russia) having a diameter of 0.5 mm. The microstructure of this wire had a strongly deformed lamellar morphology, where the grains of the polycrystalline matrix practically disappeared (**Figure 1**). The main feature of this lamellar structure is the narrow highly elongated grains collected in a bunch like a rope. As a result, deformation tracks, such as slip bands or twin lamellae, could not be revealed on the surfaces of the samples after deformation. An XRD spectrum taken from the cold drawing iridium wire prior to testing is shown in **Figure 2**. There are two high narrow peaks ((200) and (220)) in the middle angles of the spectrum taken from the sample. No visible changes in the spectrum were

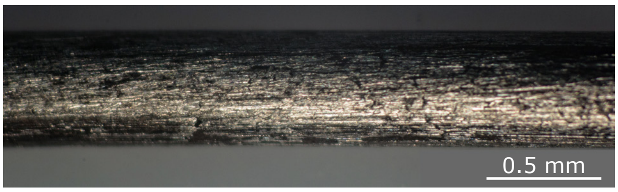

Fig. 1. Microstructure of the cold drawing iridium wire (diameter 0.5 mm)

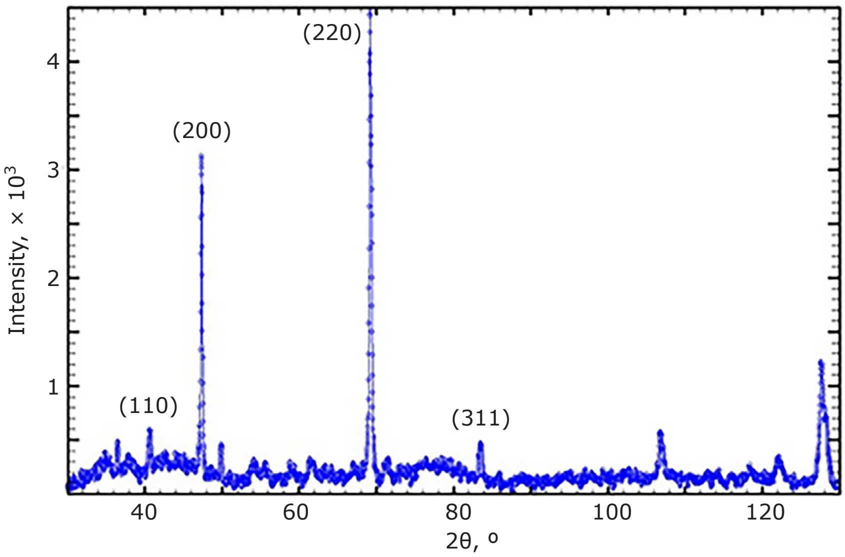

Fig. 2. XRD taken from the cold drawing iridium wire (diameter 0.5 mm)

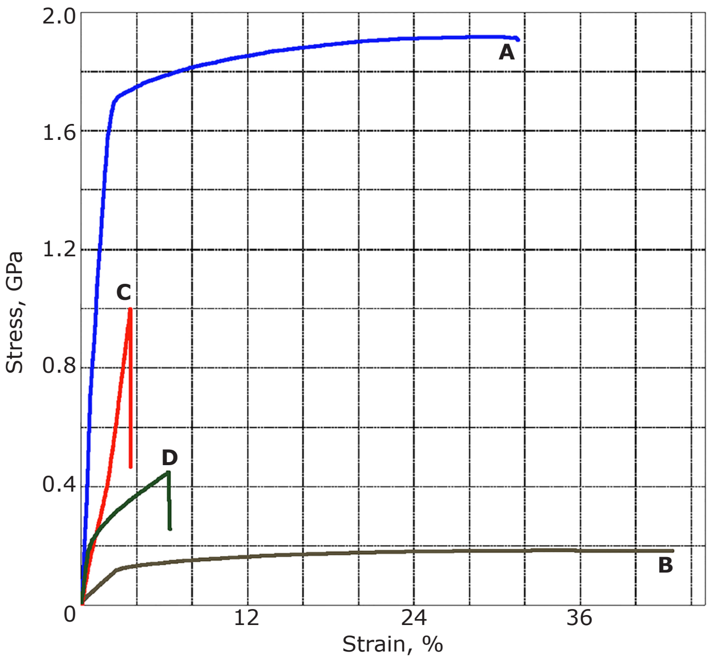

Fig. 3. Stress-strain curves under tension at room temperature: **A** cold drawing iridium wire, diameter 0.5 mm (elongation 29%, necking 23%); **B** annealed copper wire (elongation 58%, necking 55%); **C** cold drawing iridium wire, diameter 2.7 mm (elongation 3.6%, no necking); **D** cold drawing iridium wire, diameter 2 mm after recrystallisation annealing (elongation 7%, no necking)

revealed after tensile testing of the sample. It may be concluded that a stable drawing texture is formed in the iridium wire in comparison with an annealed polycrystalline sheet, which does not depend on further tensile deformation.

The second set of iridium samples consisted of 10 cold drawing wires with a diameter of 2.7 mm taken from the workpiece that was used to manufacture the thin plastic iridium wire. The Vickers microhardness of these samples in the undeformed state was about 7 GPa. The third set of iridium samples contained 10 cold drawing wires with a diameter of 2 mm. In contrast with the second set, these samples were annealed at 1000–1200°C for 20 min in a low vacuum and, as a result, their Vickers microhardness dropped up to 5 GPa. This operation is also used in the technological process for the manufacture of plastic iridium wire.

The stress-strain curves of the cold drawing iridium wires are shown in **Figure 3** and some of their mechanical characteristics are collected in **Table I**. The back surfaces of the deformed samples are shown in **Figure 4**, while their fracture surfaces are given in **Figure 5**. It is clearly visible that the deformation behaviour of the samples from the first set (**Figure 3**, curve **A**) is similar to the behaviour of annealed copper wire (**Figure 3**, curve **B**). The long stage of plastic

Table I Mechanical Properties of the Cold Drawing Iridium Wires and Annealed Copper Wires Under Tension at Room Temperature

Yield stress, $\sigma_{0,2}$, MPa	Ultimate tensile stress, σ_B, MPa	Elongation, ε, %	Thinning in neck, δ, %
Cold drawing iridium wire (0.5 mm in diameter)			
~1000	1850	30	20
Cold drawing iridium wire (2.7 mm in diameter)			
~900	1000	3.6	-
Cold drawing iridium wire (2 mm in diameter) after annealing			
~200	480	7	-
Annealed copper wire			
20	210	43	55

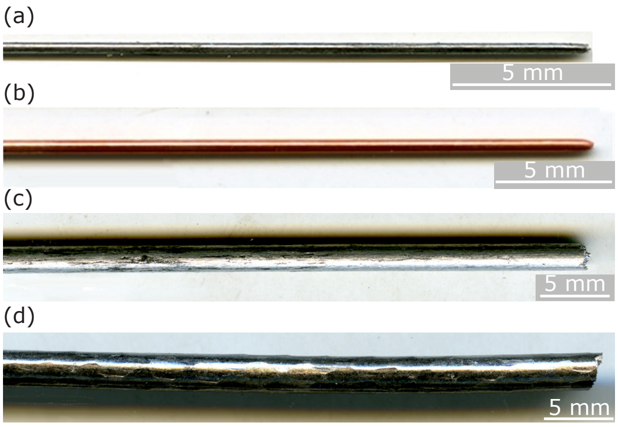

Fig. 4. Back surfaces after tensile testing at room temperature: (a) cold drawing iridium wire, diameter 0.5 mm (elongation 29%, necking 23%); (b) annealed copper wire, diameter 0.75 mm (elongation 58%, necking 55%); (c) cold drawing iridium wire, diameter 2 mm (elongation 3.6%, necking 0%); (d) cold drawing iridium wire, diameter 2.7 mm after recrystallisation (elongation 7%, necking 0%)

flow takes place after the short stage of material strengthening (**Figure 3**, curves **A** and **B**, respectively). Indeed, the total elongation of both materials may be estimated as considerable for a polycrystalline wire sample (30% for iridium and 43% for copper). In addition, there is a clearly visible advanced necking region on the back surfaces of the deformed samples (thinning of 20% for iridium and 55% for copper) (**Figures 4(a)** and **4(b)** and **Table I**). However, in contrast with copper, iridium exhibits much higher yield stress and ultimate tensile strength (**Table I**). In spite of the features that are inherent to the ductile deformation behaviour, the fracture mode of the iridium samples from the first set is attested as BTF in the strongly deformed lamellar structure (**Figure 5(a)**). The same findings were obtained for cold drawing iridium wire with a diameter of 0.3 mm in the temperature range 20–800°C in (29).

The deformation behaviour of thick cold drawing iridium wire (**Figure 3**, curve **C**) can be attested as brittle: its stress-strain curve has an almost rectilinear profile, the yield stress is similar to the ultimate tensile strength, while the deformation prior to failure is small in comparison with the previous case. No necking was observed on the back surfaces of the deformed cold drawing thick iridium wires (**Figure 4(c)**). The fracture mode of the samples agrees with their brittle behaviour, it is BTF (**Figure 5(b)**). The short-term vacuum annealing of the thick cold drawing iridium wire at a temperature close to the point of recrystallisation of iridium leads to a change in mechanical behaviour from brittle to ductile. Indeed, the behaviour of the stress-strain curve becomes similar to annealed copper (**Figure 3**, curve **D**) when after a short stage of strengthening follows the plastic flow stage, while the yield stress and the tensile strength drop considerably (**Table I**). However, its deformation prior to failure is very small (**Table I**) for a plastic material and the neck is absent in the deformed samples (**Figure 4(d)**). The fracture mode does not change from brittle to ductile: it is attested as a mixture of BTF and BIF (**Figure 5(c)**).

4. Discussion

It was shown that the deformation behaviour of cold drawing iridium wire under tension at room temperature depends on its structural state. Wire with grains of 50–100 μm behaves as a brittle material and exhibits BTF as the fracture mode. Vacuum annealing at 1000–1200°C causes a drop of yield stress of the iridium wire, but does not lead to significant increase of plasticity, while its fracture mode continues to be brittle. On the other hand, thin cold drawing iridium wire having a lamellar

(a)
(b)
(c)

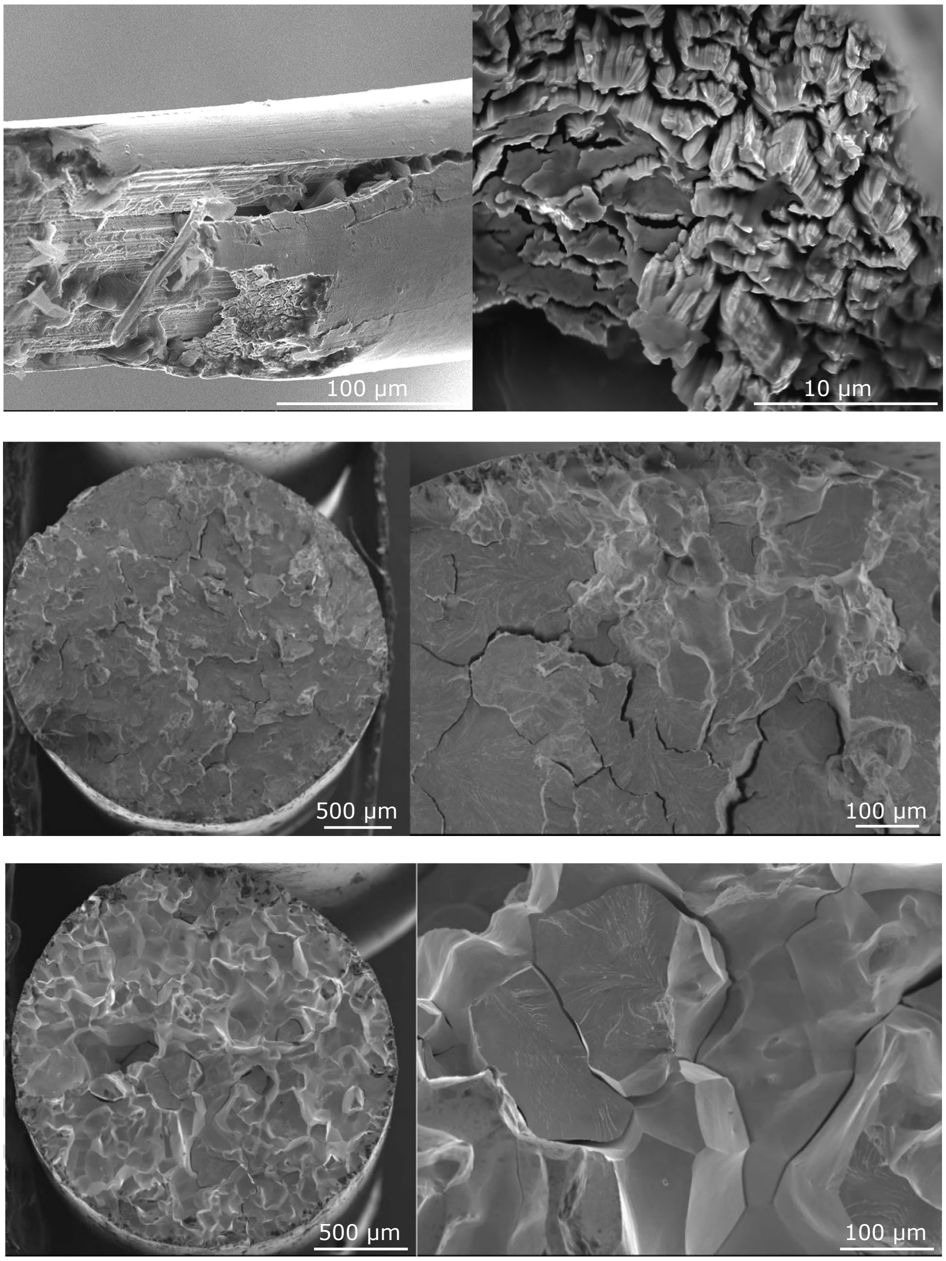

Fig. 5. Fracture surface of the cold drawing iridium wire under tension at room temperature: (a) diameter 0.5 mm (elongation 29%, necking 23%); (b) diameter 2.0 mm (elongation 3.6%, no necking); (c) diameter 2.7 mm after recrystallisation (elongation 7%, no necking)

structure demonstrates considerable elongation prior to failure and clearly visible necking, despite BTF as its fracture mode. This finding gives the basis for the conclusion that such lamellar structure is the correct morphology for plastic polycrystalline iridium. It is important to note that this morphology is formed in the iridium workpiece under the cold drawing process, while a few short terms annealing at 1000–1200°C are included in the procedure after some rolling passes (29).

Earlier, it was shown that BIF is the impurities induced fracture mode of high purity polycrystalline iridium (31, 32). Indeed such non-metallic elements as carbon and oxygen contained in a low vacuum (10^{-2} MPa) induce grain boundaries brittleness, but the kinetics of the process depends on the working temperature and its duration (31). For example, under annealing of 20 min at 1200°C, the portion of BIF on the fracture surface is considerably less than the portion of BTF, while after 24 h annealing BIF covers the whole fracture surface. It means that the regime of annealing of the cold drawing iridium wire used in this work is optimal because the hardness and the yield stress of the iridium workpiece are decreased, but the cohesion strength of grain boundaries does not drop.

The low plasticity of the thick cold drawing iridium wire, which was strongly hardened during preliminary processing, may be explained by the supposition that its resource of plasticity is finally exhausted under tension as it takes place in iridium single crystals under the same experimental conditions (26, 37). As a result, the cleavage crack can appear on any dangerous macroscopic surface defect and, hence, such wire is prone to separation by the brittle route without necking. Following this logic, the thin cold drawing iridium wire should behave the same way; however, it exhibits ductile mechanical behaviour, except its fracture mode. One cause of this puzzling effect may be the special configuration of the defect structure of iridium, whose feature on the microscopic level is a lamellar morphology. Indeed, high purity polycrystalline iridium is able to undergo severe deformation under high-pressure torsion at room temperature when the nanocrystalline structure is forming in the material (38). It is puzzling, but in this case, the surface defects play the role of the initiation of cleavage in the neck region only (29). Indeed, iridium meets some empirical cleavage criteria (18–20). However, this effect should be considered as an artefact because in contrast with other cleavable solids iridium is a plastic material in both the single crystalline and polycrystalline states and its inclination to cleavage depends on the structural state.

5. Conclusion

The lamellar structure that forms in iridium wire during the cold drawing process provides the excellent mechanical properties of polycrystalline iridium under tension: it behaves like a ductile fcc-metal excepting the brittle fracture mode. It was shown that the inclination of iridium to cleavage depends on its structural state.

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